IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of

Applicant(s) Serial No.

: Phelps, et al. : 10/625,886

Filed

: July 23, 2003

Title

: NON-TOXIC CORROSION-PROTECTION RINSES AND

Docket No.

SEALS BASED ON RARE EARTH ELEMENTS

: UVD 0299 IA / UD 268

Examiner Art Unit

: L. Zheng : 1742

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

DECLARATION OF JEFFREY A. STURGILL

Jeffrey A. Sturgill, one of the applicants in the above-identified patent application, declares as follows:

- 1. I received a B.S degree in Geology from the University of Toledo in 1986. I was employed by the University of Dayton from November 1993 until September 2006. I have been working the area of corrosion-inhibiting pigments since 1996, and in the area of corrosion/materials degradation since 1985.
- 2. I am familiar with this application as well as the Office Action mailed September 10, 2007, including the rejections made by the Examiner therein. I am also familiar with the references cited by the Examiner in that Office Action including U.S. Patent Nos. 6.200.672 to Tadokoro (treated as equivalent to WO 98/48075).
- 3. I previously prepared solutions using Tadokoro's process and the organic compounds 2-hydroxynicotinic acid, catechol, dextrose (as a surrogate for ycyclodextrin) and salicylic acid (slightly less soluble than 2-hydroxynicotinic acid) using

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the procedure set out in Tadokoro. See Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007.

- The solubility of the dextrose-containing solution was not determined because it was too high as discussed in the Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007.
- 5. The catechol/ammonium cerium IV nitrate reaction was repeated because there was not enough product (which was almost entirely pure carbon) from the earlier reaction on which to perform the solubility test. The reaction was carried out in the same way as described in Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007.
- 6. The solid reaction products prepared in accordance with Tadokoro were evaluated for their solubility characteristics. The three solid reaction products evaluated included:

 salicylic acid/ammonium cerium IV nitrate;
 act acid/ammonium cerium IV nitrate;
 and 3) 2-hydroxynicotinic acid/ammonium cerium IV nitrate.

The samples were prepared for solubility determination in a manner similar to that described in ASTM D-2448: Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment. This specification describes weighing a mass of the pigment, and then contacting the mass of powdered pigment with nine times the mass of deionized water. In the specification, the specific resistance of the 'extracting' deionized water sample placed in contact with the pigment is then determined, in order to measure how many ions were placed into the water from the pigment. This allows for a determination of the Total Salts being solubilized by the water - in effect, measuring the total solubility of the pigment in water. For this effort, that information is only part of what is needed. A measure of the cerium being extracted from the pigment/resultant solid was needed. Therefore, the extracting water sample was analyzed by inductively coupled plasma (ICP) spectroscopy in order to derive the quantity of soluble cerium in each sample.

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The ICP results for each sample are attached. Specifically, Sample 1 (reaction product of ammonium cerium IV nitrate and salicylic acid (Exhibit 1)) indicates a quantity of extracted cerium corresponding to an average of 1071 ppm (0.1 wt. %). Sample 2 (reaction product of ammonium cerium IV nitrate and catechol (Exhibit 2)) indicates a quantity of extracted cerium corresponding to an average of 6.5 ppm (0.0001 wt.%). Lastly, Sample 3 (reaction product of ammonium cerium IV nitrate and 2-hydroxynicotinic acid (Exhibit 3)) indicates a quantity of extracted cerium corresponding to an average of 81610 ppm (8.16 wt. %). As can be seen from the ICP data, smaller concentrations of other elements were detected.

Based upon a molecular weight for cerium of 140.1, these extracted concentrations correspond to cerium solubilities of:

Sample 1: 7.1 x 10-3 moles/liter

Sample 2: 4.3 x 10-5 moles/liter

Sample 3: 5.8 x 10-1 moles/liter

Tadokoro reported the solubility for the catechol and 2-hydroxynicotinic acid complexes as 0.01 mol/l or less.

- 7. The cerium content of the catechol/ammonium cerium IV nitrate reaction product is extremely low (0.0001 wt.%). The measured cerium content is probably some residual, reduced starting material. The reaction product was previously tested to be almost pure carbon. See Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007.
- 8. The portion of the extract for all three solid materials that was not used for ICP analysis was then subjected to redox determination. This analysis was previously described in Paragraph 12 of the Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007, but without the digestion procedure described because the extract was already in liquid form. Specifically, the titration was the procedure described on page 246 of Reagent Chemicals Specifications and Procedures 10th by the ACS

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Committee on Analytical Reagents, 2006. The 9:1 water/solid extract described above was titrated with a 0.1 N ferrous sulfate solution in the presence of a redox probe in order to determine if the cerium present was trivalent or tetravalent. These titration curves for Samples 1 through 3 indicate no change in oxidation state of the cerium in the presence of the ferrous reducing agent. See Exhibits 4-6. Therefore, there is no cerium (IV) present.

- Tadokoro does not describe having performed any procedure to determine the valence of the rare earth metal in the complex formed by the process described there.
- 10. The process described in Tadokoro is non-enabling for making a tetravalent cerium complex, and it would take undue experimentation to produce a tetravalent cerium complex using Tadokoro's process.

The declarant further states that the above statements were made with the knowledge that willful false statements and the like are punishable by fine and/or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent resulting therefrom.

Date: /- // - @ 8

Jeffrey A. Sturgill

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Ag 338.289	8.289	0.001625	mdd	0.001089	67.0	11.2457	1.80514 ppm	Sc 361.383		
AI 237.312	7.312	0.008277	DOM	0.003231	39.0	13.4822	9.19622 pnm	Sc 361,383		
A1308.215	8,215	0.002182uv	maa	0.003556	162.9	163.890	2.42498 mm	Sc 161.383		
AI 394.401		0.124590	שמט	0.002084	1.7	668 328	138.434 nnm	Sc 361.383		DE
A1 396 152	_	0.004138	amu.	0.000735	17.8	234 666	4 59755 mm	Sr 361 383		:C-
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Ba 455.403	5.403	0.006820	mdd	0.000114	3	7967.60	mdd 0///c/	Sc 361.383	~	Pì
Ba 493.408		0.006523	mdd	0.000145	2.2	2933.37	7.24797 ppm	Se 361.383		10
Bi 222.821		0.004332uv	undid	0.007439	171.7	6.34679	-4.81316 ppm	Sc 361.383		FS
Bi 223.061		-0.002177uv	udd	0.001594	73.3	1.57852	-2.41841 ppm	Sc 361.383		3 (
Ca 393.366		0.015970	mdd	0.000314	2.0	13380.0	17.7450 ppm	Sc 361,383	֖֖֖֝֞֝	HE
Ca 396.847		0.014858	uda	0.000286	1.9	22868.9	16.5089 ppm	Sc 361.383	<u>+</u>	ΙMΙ
Cd21	Cd 214.439	-0.000484uv	DDC	0.000101	20.8	9,43964	-0.537951 ppm	Sc 361,383		CA
Cd 22	Cd 226.502	-0.000629uv	maa	0.000206	32.7		-0.698795 nnm	Sc 361 383		LS
Cd 22		-0.000236uv	maa	0.000139	58.8		-0.261786 nom	Se 361.383		
Ce 407.570		0.961956	mou	0.004571	0.5		1068 84 nnm	Se 361.383	2	ا خ
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252	•	vac 10100.0-	udd	0.000213	21.0		-1.12/39 ppm	Sc 361.383		
Co23		-0.0000796uv	udd	0.000227	28.5		-0.884987 ppm	Sc 361.383		FA
C 20	•	-0.000166uv	undd	0.000580	348.8		-0.184609 ppm	Sc 361.383		ΧI
G 20		0.001156	urdd	0.000871	75.4		1.28401 ppm	Sc 361.383		NO.
Cr 26		-0.000689uv	udd	0.000357	51.8		-0.765230 ppm	Sc 361.383		6
Cu 2)	Cu 213.598	0.000323	urdd	0.000324	1003	2.87198	0.359328 ppm	Sc 361.383		14
Cu 32	Cu 324.754	0.000727	udd	0.000214	29.4	74.8020	0.807281 ррт	Sc 361.383		22
Cu 32	Cu 327.395	0.002441	udd	0.001159	47.5	90.2475	2.71271 ppm	Sc 361.383		511
Fe 23	Fe 234.350	0.013650	undd	0.000709	52	56.9161	15.1663 ppm	Sc 361,383	7	175
Fe 23	Fe 238.204	0.011741	undd	0.000072	9.0	143.163	$13.0450 \mathrm{ppm}$	Sc 361.383	2	
Fe 25	Fe 259.940	0.012032	undd	0.000198	1.6	103,599	13.3690 ppm	Sc 361,383		
Hg 18	Hg 184.887	0.001055	mdd	0.001188	112.6	1.27863	1.17198 ppm	Sc 361.383		
Hg 19	Hg 194.164	0.001115	mdd	0.000707	63.4	2.2223	1.23879 ppm	Sc 361.383		
Hg 2	Hg 253.652	0.000512uv	mdd	0,000217	42.3	5.67644	-0.568908 ppm	Sc 361.383		
K 766.491	5.491	0.001388	mdd	0.001041	75.0	569.479	1.54171 ppm	Sc 361.383		
K 76	K 769.897	0.012038	mdd	0.004695	39.0	14527.6	13,3753 ppm	Sc 361,383		
Li 61	Li 610,365	0.000979	mdd	0.000652	66.5	50.8482	1.08819 ppm	Sc 361.383	1	
1167	Li 670.783	0.002479	mdd	0.000032	13	14236.6	2.75453 ppm	Sc 361.383		EVHIBII
Mg 2	Mg 279.553	-0.001352uv	mad	0.000018	1.3	606.081	-1.50184 ppm	Sc 361.383	poleg	-
Mg 2	Mg 280.270	-0.000974uv	TI CO	0.000018	1.8	156.918	-1.08170 ppm	Sc 361,383	evo.	
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7.7	7 -	2.6	3952.8	40.8	83.8	100.8	44.7	53.7	82.6	30.7	9.9/	73.7	16.8	37.3	10.5	445.4	34.1	90.3	34.1	23.4	31.9	91.1	558.0	66	53	9.4	30.5	4.8	35.4	26.4	9.0	1.2	101.5	190.1	49.1	29.5	36.4	35.3	2.2	3.6	0.8	4.5	7.9	17 9
0.000693	0.00083	0.001794	0.001088	0.000185	0.000677	0.000828	0.004561	0.003880	0.015168	0.001670	0.001836	0.006788	0.001310	0.000571	0.003645	0.003857	0.001701	0.003177	0.021959	0.014581	0.003626	0.008235	0.004832	0.003621	0.001823	0.003222	0.001762	0.002181	0.003098	0.000324	0.000012	0.000021	0.000151	0.002502	0.000735	0.015929	0.019152	0.017152	0.000170	0.000269	0.000066	0.000266	0.000301	ひ のののならら
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-0.009045uv	0.065301	0.068574	-0.000028uv	-0.000454uv	-0.000807uv	-0.000821uv	0.010203	0.007218	0.018367	-0.005539uv	0.002398	-0.009214uv	-0.007789uv	0.001531	0.034819	~0.000866uv	-0.004993uv	0.003517	0.064413	0.062306	0.011364	0.009036uv	-0.000866uv	0.036433	0.034397	0.034174	0.005784	0.045012	-0.008765uv	-0.001224uv	-0.001985uv	-0.001787uv	-0.000149uv	0.001316uv	0.001496	0.054019	0.052646	0.048642	0.007622	0.007490	0.008242	0.005855	0.003817	O 00355R
Mo 204.598 Mo 284 824	Na 588.995	Na 589,592	Ni 216.555	Ni 221.648	Ni 230.299	Ni 231.604	P 177.434	P 213.618	P 214.914	Pb 182.143	Pb 220.353	Pb 283.305	Pd 229.651	Pd 340.458	Pd 360.955	Pt 177.648	Pt 203.646	Pi 214.424	S 180.669	S 181.972	Sb 206.834	Sb 217.582	Sb 231.146	Si 250.690	Si 251.611	Si 288.158	Sn 189.927	Sn 235.485	Sn 283.998	Sr 216.596	57.401.771	Sr 421.552	11 554 941	11 336.122	11.537,280	W 207.912	w 209.475	W 220.449	Zn 202.548	Zn 206.200	Zn 213.857	Zr 339.198	Zr 343.823	Zr 349.619

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Sb 206.834 Sb 217.582 Sb 231.146

Pt 203.646

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Pt 177.648 Pt 214.424 89.8 90.1 45.9 38.6 97.4 35.5 66.4 38.9 0.4 19.2 9

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3b 182.143 Pb 283,305 Pd 229.651 Pd 340.458 Pd 360,955

P 177.434 P 213.618 P 214.914 Pb 220.353

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Sn 189.927 Sn 235.485 Sn 283.998 Sr 216.596 Sr 421.552 Fi 337.280 W 207.912 W 209.475

3r 407.771 Fi 334.941 Ti 336,122

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1069.82 92.2989 SD(int) %RSD 692.988 1.3 1182.709 1.4 416.957 2.0 843.698 1.3

203859 4 65728.8 6 85910.1 11 20385.9 4 65728.6 8

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Label Sc 335,372 Sc 336,383 Sc 363,074 Sc 424,682

Sc 361.383	1.00838	54551.8 85910.1	54551.8 692.988 85910.1 1182.709		NE 4			
Sc 363.074	1.00295	20385.9	416.957	50				
Sc 424.682	1.02635	65728.6	843.698		E			
3- JS (Samp)	12	(2/10/2007, 11:50:38 AM	0:38 AW	Ē	Tube 19			
Weight: 0.1	A	Volume: 100		Ď	Dilution: 1	٠		
	Sol'n Cone.	Units	CS	%RSD	Int. (c/s)	SD %RSD Int. (c/s) Cale Cone	SI 19 300	
Ag 328.068	0.026105) udd	0.001287	4.9	358.221	26.1050 mm	Sc 361 383	
Ag 338.289	0.025171) udd	0.000754	3.0	43,3597	25.1714 ppm	Se 361 383	
AI 237.312	0.028340	_	0.003601	12.7	26.4114	28.3397 nnm	Se 361 383	
AI 308.215	0.057203	bbm	3.000675	1.2	296.182	57.2029 npm	Se 361 383	
Al 394.401	10.8704x	_	0.015898	-0	53876.8	10870.4 ppm	Sc 361.383	
AI 396.152	0.103606	_	0.014992	14.5	2096.74	103,606 ppm	Se 361 383	
As 188.980	-0.009494uv	_	0.005764	60.7	1.49754	-9.49421 ppm	Sc 361 383	
As 193.696	-0.007416uv	Ī	0.004732	63.8	0.687385	-7.41614 nnm	Se 361 383	
As 234.984	0.210811) mdd	0.015360	7.3	35.5309	210.811 ppm	Sc 361.383	
B 208.956	0.024253) mdd	0.003583	14.8	7.04414	24.2528 ppm	Sc 361.383	
B 249.678	0.026916) undd	0.000854	3.2	99.3344	26.9164 ppm	Sc 361.383	52
B 249.772	0.029638) udd	0.000645	7.7	212.957	29.6380 ppm	Se 361.383	
Ba 233.527	-0.000854uv) udd	0.000265	31.0	12.9036	-0.853843 ppm	Se 361.383	
Ba 455.403	-0.001370uv) mdd	0.000041	3.0	360.875	-1.37040 ppm	Sc 361.383	
Ba 493.408	-0.000271uv) undd	0.000091	33.5	756.516	-0.270828 ppm	. Sc 361.383	
Bi 222.821	-0.016004uv		0.005075	31.7	3.88297	-16.0039 ppm	Sc 361.383	
19773.061	0.003142		0.002084	663	4.92885	3.14230 ppm	Sc 361.383	
Ca 393.366	0.143266		0.000575	9.4	83101.0	143.266 ppm	Sc 361.383	
Ca 396.847	0.007374		0.000108	1.5	15119.3	7.37360 ppm	Sc361.383	,
Cd 214.439	0.000144		0.000118	81.9	14.2006	0.144496 ppm	Sc 361.383	
705.2020	0.001022		0.000111	8.0	31.6891	1.02227 ppm	Sc 361.383	
Ce 407 570	91 0450		0.000218	27.	31.1482	1.71449 ppm	Sc 361.383	7 51.16
Ce 418 659	80 8462v		1.071040	3 6	001097	81045.0 ppm	Sc 361.383	4
Ce 446.021	82.9391x	The state of	760779	2 0	581908	82030 1 mm	50 301.383	19 TO 1
Co 228.615	0.001210		0.000693	573	9 54536	1 20073 ppm	50,101,363	ا ا
Co 230.786	0.004663		0.000190	4	23 5778	mdq 6753-1	Sc 261 202	
Co 238.892	-0.000740uv		0.000056			mdd CC200.1	60 161 303	
Cr 205.560	0.008068		0.000508			8 06833 mm	Sc 261 202	
Cr 206.158	0.007592	_	0.000722	9.5	5.06906	7.59248 ppm	Sc 361 383	
Cr 267.716	0.005238	_	0.000170	33	66.5452	5.23759 mm	Se 361.383	
Cu 213.598	0.014996	_	3.000718	4.8	29.4406	14.9956 ppm	Sc 361.383	
Cu 324.754	-0.001975uv) udd	0.000110	5.6	46.6102	-1.97452 ppm	Sc 361.383	cr)
222 206	, 2000 0							

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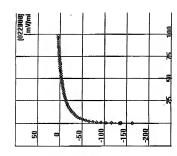
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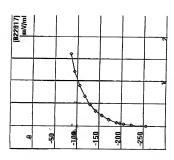
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Sc 361.383 Sc 361.383	Sc 361.383	Sc 361.383	Sc 361 383	Sc 361.383	Sc 361.383	Se 361.383	Sc 361,383	Sc 361.383	Sc 361.383	Sc 361.383	Se 361.383	Sc 361.383	Sc 361.383	56 301.383	50.101.383	Sc 161 383	Sc 261 203	Sc 361 292	Se 261 202	Sc 361 383	Se 361 383	Se 361 383	Se 361 383	Sc 361.383	Sc 361.383	Se 361,383	Sc 361,383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361 383	Sc 361.383	Sc 361.383	Sc 361.383	Se 361 383	Cr 175 72
	-1.12060 ppm	2 13314 npm	0.072558 ppm	6.67586 ppm	2.19974 ppm	4.04404 ppm	-0.286297 ppm	0.184040 ppm	1.98100 ppm	-9.09147 ppm	-7.91447 ppm	-3.07109 ppm	32.2368 ppm	71 22 Ppm	1 18582 mm	4 18806 nom	-0 105737 mm	36 8598 nnm	5 87969 nnm	23.1145 ppm	36.3252 ppm	35.7528 ppm	-14.3803 ppm	-25.6118 ppm	80.7439 ppm	3056.31 ppm	0.092131 ppm	397.415 ppm	11.8510 ppm	-32.1826 ppm	-12.4820 ppm	0.837360 ppm	4.13309 ppm	85.7720 ppm	308.348 ppm	293.107 ppm	364.334 ppm	56.7237 ppm	83.6487 ppm	38.8928 прш	-1.19299 npm	3 58799 nnm
3.43383 -2.32983	449.47]	105 009	5616.92	2263.75	328.486	91.7467	74.2138	65.7486	61.0384	4.32455	5.28948	21.2989	1/190.3	41 0020	986791	15.0690	4.05582	3.60943	4.33868	2.36318	3.54721	30.1130	32.5731	-6.88811	295.645	5707.18	1.06155	55.4158	6.91495	9.89024	13.1173	1.72372	1.71475	24.9653	327.665	375.631	944.874	9.26087	14.8247	37.8579	1.45497	8141 RD
31.3	79.6	50.5	46.5	1.5	3.8	4.2	14.8	61.8	12.3	2.7	6 6	13.0	6.0	3 -	27.6	10.0	358.5	4.6	17.2	619	11.1	10.5	40.2	23.0	4.7	0.2	7629.9	9.0	25.6	23.2	165.8	744.8	155.5	8.	17	0.8	0.2	6.9	9.0	113	13.0	1.7
0.000910	0.000892	0.001077	0.000034	0.000099	0.000085	0.000172	0.000042	0.000114	0.000243	0.000249	0.000735	0.000416	0.001913	0.000003	0.000327	0.000418	0.000379	0.001690	0.001011	0.014303	0.004048	0.003744	0.005781	0.005900	0.003781	0.007109	0.007029	0.002561	0.003033	0.00/466	0.020699	0.002049	0.006428	0.004125	0.00367	0.002292	0.000672	0.003912	0.007522	0.004385	0.000155	0.000061
udd	ondid.		bbu	udd	ppm	udd	шdd	udd	udd	bbu	mdd.	H		E 00	000	mdd	udd	wdd	mdd	udd	udd	mdd	udd	mdd	undid	mdd	mdd	E dd		Edd		The last	EL I	E.	mdd	mdd	mdd	undd	mdd	udd	mdd	maa
0.002343	-0.001121uv	0.002133	0.000073	9.006676	0.002200	0.004044	-0.000286uv	0.000184	0.001981	-0.00909 Juv	-0.007914uV	0.032237	0.044784	0.021337	-0.001186uv	0.004188	-0.000106цу	0.036860	0.005880	0.023115	0.036325	0.035753	-0.014380uv	-0.025612uv	0.080744	3.05631x	0.000092uv	0.397415	1001100	-0.032103UV	0.000837	0.000122	0.004155UV	0.000772	0.500.90	0.293107	0.364334	0.056724	0.083649	0.038893	-0.001193uv	0.003588
Hg 253.652	K 769.897	Li 610.365	Li 670.783	Mg 279.553	Mg 280.Z/0	Mg 285.213	010.7.62 mM	Me 204.572	Me 200 02	Mo 204 509	Mo 284 824	Na 588 995	Na 589,592	Ni 216.555	Ni 221.648	Ni 230,299	Ni 231.604	P 177.434	P 213.618	P 214.914	Pb 182,143	Pb 220.353	Pb 283,305	Pd 229.651	Pd 340.458	Fu 300.353	Pt 202 646	Pt 214 434	S 180 660	S 181 972	Sb 206.834	Sh 217 582	Sb 231 146	Si 250 690	00000000	5:290 150	St 266.138	/76.481 nc	Sn 233.485	St. 285.998	Sr 216.596	Sr 401.771

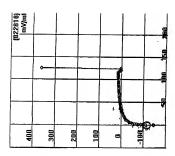
		ይ ሚሚኝነ	SHS:	SD(In) 133.442 316.612 92.365	52511.3 92339.8 18727.9	Rafie 0.970661 1.09021 0.921376	abet 25 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5
Sc 361.383	43.2557 ppm	1031.43	2.6	0.001119	udd	0.043256	£r 349.619
Sc 361.383	23.0793 ppm	543,630	1.7	0.000393	urdd	0.023079	Sr 343.823
Sc 361.383	6.59574 ppm	34,3309	3.2	0.000211	mdd	0.006596	r 339.198
Sc 361.383	1.97315 ppm	24.7303	6.7	0.000132	mdd	0.001973	Zn 213.857
Sc 361.383	7.31213 ppm	8.32183	3.0	0.000216	mdd	0.007312	Za 206.200
Sc 361.383	5.46624 ppm	29.0883	3.9	0.000212	mdd	0.005466	Zn 202,548
COCTACAC	midd ozowar-	00000000	3	**	AA		4411



REDOX TITRATION
OF 9:1 Extract
OF (NH,) 2Ce (MO3)e
SALICYLIC AIR.10
RESECTION PRODUCT







OF (NH4)2 CE (NO3)6)
NIGTHICS
2-RYDEOXY WARTHER AC REDOK TITRATION OF 9:1 FEXTRACT REACTION PRODUCT

